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The Analysis of Effect on the Heat Conduction Oil Smoke Ingredients by Different Ignition Ways

Liu He[a,*], Jie Yang[a]

The Armed Police Forces Academy, Langfang 065000, China

Abstract

This article analyzes the smoke ingredients of heat conduction oil by Gas Chromatography-Mass Spectrometer (GC-MS).Meanwhile, the effect on smoke dust ingredients by two different ignition ways of touching high-temperature surface and wick will be compared. Moreover, the different influences on the smoke by diverse extracts are studied as well. The result shows that n-hexane has better effect than aether. Heat conduction oil produces more polycyclic aromatic hydrocarbon (PAH) by the way of touching high-temperature surface and it produces more oxidizing materials by the other way. The research can provide a certain assistance for the analysis of the combustion of the heat conduction oil in fire physical evidence identification.

Peer-review under responsibility of the organizing committee of ICPFFPE 2015

Keywords: Heat conduction oil ; Smoke dust ; Gas Chromatography-Mass Spectrometer ; Physical evidence identification

1. Experiments

Heat conduction oil is an excellent kind of heat carrier with characters like thermostability, high heat efficiency and easy temperature control. It is widely used in petrochemical engineering, building material, food processing, plastics, paper industry and so on. However, it belongs to combustible organics and will lose performance and value. What's worse, it will have a danger of fire and explosion due to the thermal cracking and polycondensation which probably happen during using [1-2]. On the other hand, it will affect combustion adjuvant's test like gasoline during conflagration evidence identification and the cognizance's accuracy of arson. The GC-MS technology combines high separation efficiency of gas chromatography with correct confirming the compound's structure of mass spectrometer [3-4]. So it is widely used in conflagration evidence identification. We will successfully analyze the smoke ingredients and distinguish heat conduction oil from gasoline. At the same time, the proper extract is prerequisite. The samples extracted from fire scene are dust or residues and we choose the residues with dust as samples because the fire ground is so complex that it is difficult to extract samples [5]. In the same condition such as color spectrum, mass spectra and samples etc. The effect on smoke dust ingredients by two ignition ways of high temperature surface and wick will be compared. Besides, the effect on the GS-MC's result of the heat conduction oil smoke ingredients by three kinds of extracts, absolute ether, n-hexane, and the mixture of both will be contrasted.

* Corresponding author. Tel.: +86-13952060413
E-mail address: emma-heliu@qq.com

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2. Experiments

2.1 Equipment and Material

Equipment: Agilent 6890/5973 N GC-MS (Agilent Technology Inc.); G1033A,D01.00NIST08.L Mass spectral database ; HP-5MS chromatography column (30m*0.25mm*0.25μm).

Material: Mobil 603 Mineral Heat Conduction Oil.

Extracts: n-hexane, absolute ether, mixture of n-hexane and absolute ether.

2.2 Sample Preparation

2.2.1 Ignited by High-temperature Surface

First, put the crucible into the close roaster, heat it for 10 min from room temperature to 800℃ ， and keep the temperature for 20 min before put it out. Then use the igniter to light it after pour the 800℃ heat conduction oil on the carrier surface rapidly. Meanwhile, put a piece of glass 15cm above the crucible to let the smoke be sorbed on the glass. Use 3 pieces of absorbent cotton to collect the smoke dust, then put them into 10 mL n-hexane, 10 mL aether and 10 mL aether of both. Stir them completely for 30 min, then concentrate statically after extract again and again. At last, filter them for constant volume and put them into centrifuge tube for use.

Sample 1: MF-n-hexane; Sample 2: MF-aether; Sample 3: MF-mixture of n-hexane and aether.

2.2.2 Ignited by Wick

First, put 10 mL heat conduction oil into the crucible, and then soak the 2 cm wick with the oil. After that , make one end of the wick under the level of oil and the other part of oil .Use the igniter to light the upper part , and the same time , put a piece of glass 15 cm. Above the crucible to let the smoke be sorbed on the glass . Use a piece of absorbent cotton to collect smoke dust, then put it into 10 mL mixture of n-hexane and aether. Stir it for 30 min, concentrate statically after extract again and again. At last, filter it for constant volume and put it into centrifuge tube for use.

Sample 4: DX-mixture of n-hexane and aether.

2.3 Condition Analysis

2.3.1 Chromatographic Conditio

The carrier gas is He (purity 99.999%). The velocity of carrier gas is 1.2 mL • min^{-1} . The pressure before column is 10 pis. The split ratio is 10:1.

We use the way of step rasing as for the programme of column temperature. The initial temperature is 50℃ . Keep it for 2 min. Then keep the way of rasing temperature to 150℃ by the speed of 10℃*min-1 for 2min. Then keep the way of rasing temperature to 250℃ by the speed of 6℃ • min-1 for 10min .

2.3.2 Mass Spectrometry Condition

GC-MS the temperature of interface: 280℃; the temperature of ion source: 230℃; the temperature of quadrupole: 150℃.

El ion source, electronic energy: 70 eV.

The mass range of full scan: 50~500 amu.

To protect the filament, set the delayed time of solvent 3 min.

3. Results and Discussion

3.1 The Research on the Effect on the Oil Smoke Ingredients by different Extracts

After analyzing sample 1, 2, 3 by GC-MS, we get the chromatography spectrogram of total ion source, see Fig 1, and the chromatographic peaks are unanimous. By automatic integration, we get the number of the chromatographic peaks which can be detected together with their preserved time and the area of the peak. By screening peaks whose match degree is

higher than 75, conclusion comes that we detect 18 materials from Sample 1, see Table 1. We detect 8 materials from Sample 2, see Table 2. We detect 31 materials from Sample 3, see Table 3.

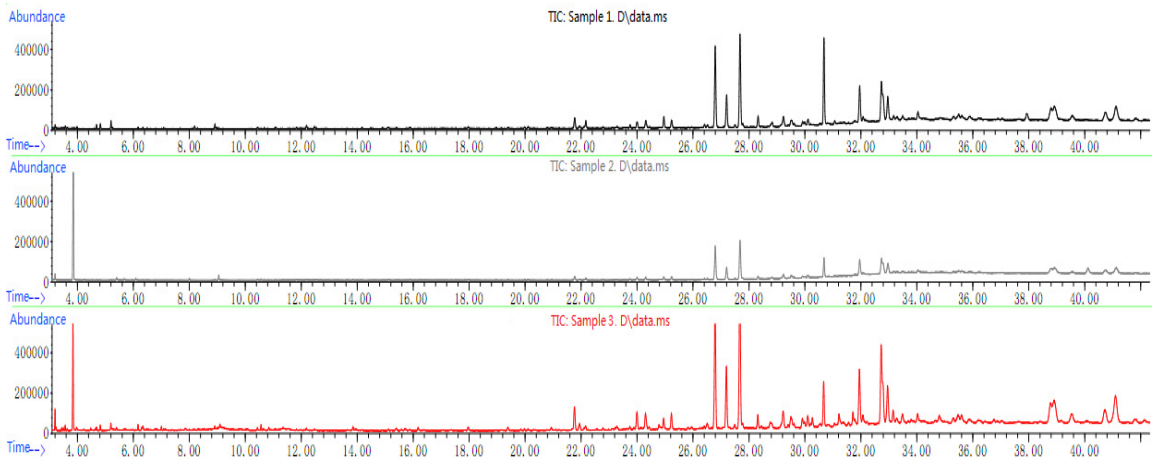
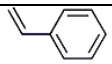
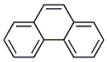
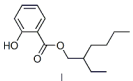
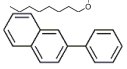
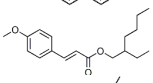
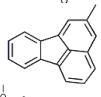
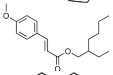
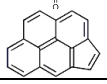


Fig.1 The chromatogram spectrogram of total icon beam detected by GC-MS from Sample 1, 2, 3

Table 1. The parameter of the peak chosen by GC-MS from sample 1(MF-n-hexane)

Number	Retention Time (t_R·min^{-1})	Molecular Tormula	Structural Formula	Peak Area	Percentage Composition （%）
1	5.204	C_8H_8		538149	0.57%
2	21.773	$C_{14}H_{10}$		1632634	1.73%
3	22.174	$C_{15}H_{22}O_3$		1289686	1.36%
4	24.961	$C_{20}H_{30}O_4$		1676561	1.77%
5	25.236	$C_{16}H_{12}$		1297708	1.37%
6	26.795	$C_{16}H_{10}$		12890730	13.63%
7	27.681	$C_{16}H_{10}$		14853175	15.70%
8	28.329	$C_{18}H_{26}O_3$		1543847	1.63%
9	29.235	$C_{17}H_{12}$		2161337	2.28%
10	30.682	$C_{18}H_{26}O_3$		11931960	12.61%
11	32.738	$C_{18}H_{10}$		11528367	12.19%

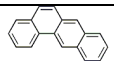
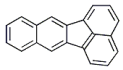
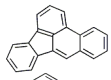
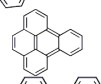
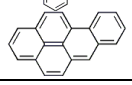
12	32.963	$C_{18}H_{12}$		4825237	5.10%
13	38.799	$C_{20}H_{12}$		3569470	3.77%
14	38.915	$C_{20}H_{12}$		5387144	5.69%
15	40.748	$C_{20}H_{12}$		2963416	3.13%
16	41.121	$C_{20}H_{12}$		5272224	5.57%

Table 2. The parameter of the peak chosen by GC-MS from Sample 2(MF-aether)

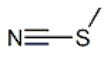
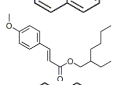
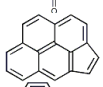
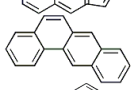
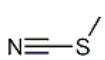
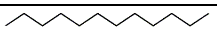
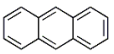
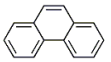
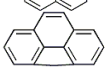
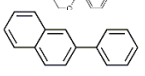
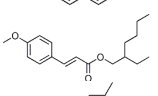
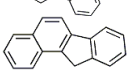
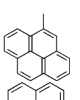
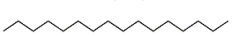
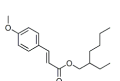
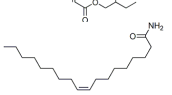
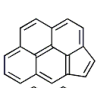
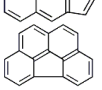
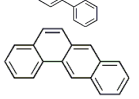
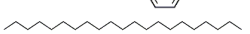
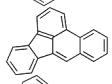
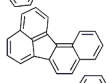
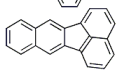
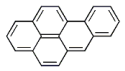
Number	Retention Time (t_R·min^{-1})	Molecular Tormula	Structural Formula	Peak Area	Percentage Composition （%）
1	3.204	C7H8		466835	1.25%
2	3.858	C_2H_2NS		13971763	37.52%
3	26.796	$C_{16}H_{10}$		5186883	13.93%
4	27.683	$C_{16}H_{10}$		6117846	16.43%
5	30.684	$C_{18}H_{26}O_3$		2518458	6.76%
6	32.740	$C_{18}H_{10}$		2829236	7.60%
7	32.789	$C_{18}H_{12}$		1667185	4.48%
8	32.969	$C_{18}H_{12}$		2189468	5.88%
9	41.130	C20H12		2293665	6.16%

Table 3. The parameter of the peak chosen by GC-MS from Sample 3(MF-mixture of n-hexane and aether)

Number	Retention Time (t_R·min^{-1})	Molecular Tormula	Structural Formula	Peak Area	Percentage Composition （%）
1	3.204	C_7H_8		1154553	0.69%
2	3.844	C_2H_2NS		8430570	5.02%
3	5.198	C_8H_8		487938	0.29%

4	10.566	$C_{12}H_{26}$		462689	0.28%
5	21.950	$C_{14}H_{10}$		1324729	0.79%
6	21.766	$C_{14}H_{10}$		3855333	2.29%
7	24.000	$C_{13}H_8O$		2674321	1.59%
8	24.304	$C_{15}H_{10}$		2754882	1.64%
9	24.954	$C_{16}H_{22}O_4$		1429572	0.85%
10	25.230	$C_{16}H_{12}$		2519888	1.50%
11	26.792	$C_{16}H_{10}$		20354106	12.11%
12	27.675	$C_{16}H_{10}$		23702744	14.10%
13	28.319	$C_{18}H_{26}O_3$		1685421	1.00%
14	29.224	$C_{17}H_{12}$		3673470	2.19%
15	29.505	$C_{17}H_{12}$		1993004	1.19%
16	29.983	$C_{17}H_{12}$		1092970	0.65%
17	30.103	$C_{17}H_{12}$		2089958	1.24%
18	30.268	$C_{16}H_{34}$		1503670	0.89%
19	30.671	$C_{18}H_{26}O_3$		6512241	3.88%
20	31.220	$C_{18}H_{35}NO$		1822264	1.08%
21	31.718	$C_{20}H_{42}$		1049647	0.62%
22	31.948	$C_{18}H_{10}$		9762141	5.81%
23	32.726	$C_{18}H_{10}$		20811524	12.38%
24	32.957	$C_{18}C_{10}$		7790892	4.64%
25	33.153	$C_{18}H_{12}$		2508074	1.49%
26	33.489	$C_{21}H_{44}$		2142383	1.27%

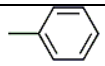
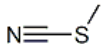
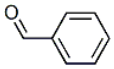
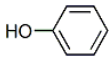
27	34.028	$C_{17}H_{10}O$		1590003	0.95%
28	38.773	$C_{20}H_{12}$		5809336	3.46%
29	38.896	$C_{20}H_{12}$		3565394	2.12%
30	38.914	$C_{20}H_{12}$		4947725	2.94%
31	39.531	$C_{20}H_{12}$		3309683	1.97%
32	40.726	$C_{20}H_{12}$		4659495	2.77%
33	41.102	$C_{20}H_{12}$		10579804	6.30%

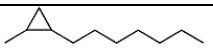
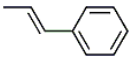
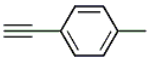
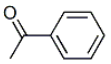
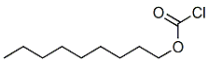
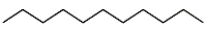
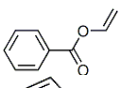
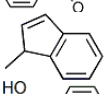
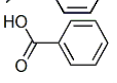
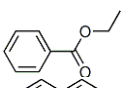
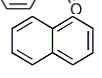
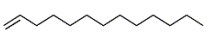
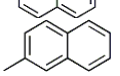
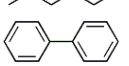
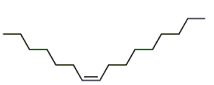
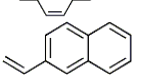
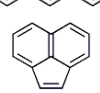
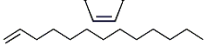
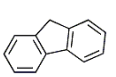
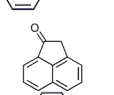
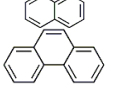
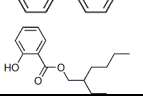
According to these tables, the materials detected from Sample 1, 2 can be detected by Sample 3, except as some plasticizers. And we can detect some specific materials from Sample 3. Among all the materials, Fluoranthene, Pyrene, Cyclopenta[cd]pyrene, Benz[a]anthracene and Benzo[e]pyrene can be detected by Sample 1, 2, 3. Styrene, Anthracene, Naphthalene, 2-phenyl-, Fluoranthene, 2-methyl-, Benz[e]acephenanthrylene, Benzo[k]fluoranthene and Benzo[a]pyrene can be detected by Sample 1, 3. Toluene, Thiocyanic acid, methyl ester, Triphenylene, can be detected by Sample 2, 3. Dodecane, Phenanthrene, 1H-Phenalen-1-one, 4H-Cyclopenta[def]phenanthrene, Benzo[a]fluorine, Pyrene, 4-methyl-, Pyrene, 1-methyl-, Hexadecane, 9-Octadecenamide, (Z)-, Tetracosan, Benzo[ghi]fluoranthene, Heneicosane, Benz[de]anthracen-7-one, Benzo[j]fluoranthene and Perylene can be detected only by Sample 3. Thus, the mixture have best extract effect to oil smoke, the n-hexane second, the absolute ether least.

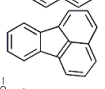
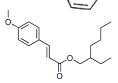
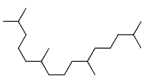
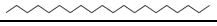
3.2 The Research on the Effect on the Oil Smoke Ingredients by different Ingnition

After analyzing Sample 4 by GC-MS, we get its chromatogram spectrogram of total ion source. We detected 40 materials, see Table 4.

Table 4. The parameter of the peak chosen by GC-MS from Sample 4(DX-mixture of n-hexane and aether) *Author Artwork*

Number	Retention Time (t_R·min^{-1})	Molecular Tormula	Structural Formula	Peak Area	Percentage Composition （%）
1	3.211	C_7H_8		1134252	1.10%
2	3.839	C_2H_2NS		1141977	1.11%
3	4.688	C_8H_{10}		527767	0.51%
4	4.823	C_8H_{10}		628187	0.61%
5	5.202	C_8H_8		1072797	1.04%
6	6.471	C_7H_6O		836130	0.81%
7	6.777	C_6H_6O		1082854	1.05%

8	6.998	$C_{11}H_{22}$		481219	0.47%
9	7.092	C_9H_{10}		404603	0.39%
10	8.016	C_9H_8		928924	0.90%
11	8.384	C_8H_8O		958196	0.93%
12	8.770	$C_{10}H_{19}ClO_2$		830186	0.81%
13	8.913	$C_{11}H_{24}$		834323	0.81%
14	9.572	$C_9H_8O_2$		1698363	1.65%
15	9.837	$C_{10}H_{10}$		1018597	0.99%
16	10.029	$C_7H_6O_2$		21219807	20.65%
17	10.155	$C_9H_{10}O_2$		972824	0.95%
18	10.433	$C_{10}H_8$		10670303	10.38%
19	11.989	$C_{13}H_{26}$		1155025	1.12%
20	12.183	$C_{11}H_{10}$		2286872	2.22%
21	12.468	$C_{11}H_{10}$		2780947	2.71%
22	13.640	$C_{12}H_{10}$		6191786	6.02%
23	13.696	$C_{14}H_{28}$		908789	0.88%
24	14.663	$C_{12}H_{10}$		1429400	1.39%
25	15.117	$C_{12}H_8$		6614734	6.44%
26	15.740	$C_{13}H_{26}$		745318	0.73%
27	17.842	$C_{13}H_{10}$		1354650	1.32%
28	19.384	$C_{12}H_8O$		993971	0.97%
29	21.760	$C_{14}H_{10}$		3488494	3.39%
30	22.159	$C_{15}H_{22}O_3$		1426261	1.39%

31	23.268	$C_{16}H_{22}O_4$		1333146	1.30%
32	24.939	$C_{16}H_{22}O_4$		1777398	1.73%
33	26.775	$C_{16}H_{10}$		1407975	1.37%
34	27.662	$C_{16}H_{10}$		798156	0.78%
35	30.661	$C_{18}H_{26}O_3$		9932222	9.66%
36	33.148	$C_{25}H_{52}$		1965157	1.91%
37	34.016	$C_{20}H_{42}$		2732953	2.66%
38	35.404	$C_{18}H_{38}$		116862	0.11%
39	35.884	$C_{18}H_{37}I$		3775459	3.67%
40	37.599	$C_{20}H_{42}$		3124200	3.04%

According to the chromatogram spectrogram of total icon beam detected by GC-MS from Sample 3 and 4, see Fig 3 and 4, we learn that the retention time of existing peaks from sample 3 appears 20 min later. We learn that the retention time of existing peaks from sample 4 appears 25 min ago.

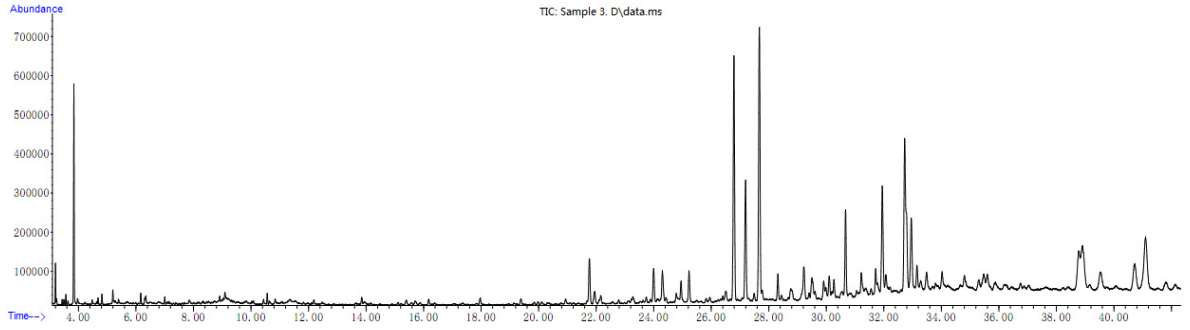


Fig 3. The chromatogram spectrogram of total icon beam detected by GC-MS from Sample 3(MF-mixture of n-hexane and aether)

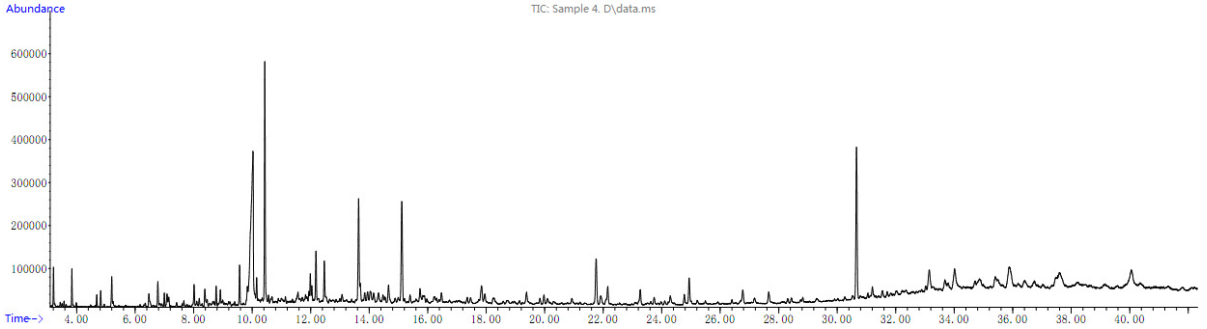


Fig 4. The chromatogram spectrogram of total icon beam detected by GC-MS from Sample 3(DX-mixture of n-hexane and aether)

By the integration of the spectrogram, we choose the peak that can be easily analyzed in appearances. By G1033A,D01.00NIST08.L Mass spectral database, we get the area of automatically integrated peak. Thereby, we get the percentage composition of each materials. Whatever the light condition is, the oil smoke is made up of fatty hydrocarbon, substituted benzene, PAH, oxygen carrier and others. The smoke ignited by heat surface has the corresponding percentage composition of 3.06%, 0.98%, 81.58%,14.37%. While The smoke ignited by wick has the corresponding percentage composition of 11.37%, 4.55%, 37.01%， 46.68% . The increase of oxygen shows that the process that the wick lights the oil let more oxygen have reactions with the oil.

4. Conclusion

In summary, n-hexane as extracts has better effect than aether. However, it will make the deficiency of some composition. So we suggest mixture of n-hexane and aether for extracting. It is caused by being ignited by heat surface, which the retention time of existing peaks appears 20 min later and it produce more PAH. It is caused by being ignited by the wick, which the retention time of existing peaks from appears 25 min ago and it produce more oxygen carrier.

Because the research on heat conduction oil analysis by GC-MS has no standard for reference, the basis that we use the peak whose match degree higher than 75 to compare the extracts effect is not perfect. While, this research will be a reference for the analysis of heat conduction oil by GC-MS in a certain extent. And we can infer the ignition of the oil by the method of percentage composition. It will offer a certain help to fire physical evidence identification.

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